Notes

Some notes on the direct current discharge detector

The direct current low pressure discharge type detector for gas chromatography reported by FISHER AND MCCARTY[1] has been of great interest for the sensitive detection of carbon dioxide and carbon monoxide.

In a series of experiments from which this note has been prepared, a detector which was similar in construction to that described by FISHER AND MCCARTY was used. There were two alterations: first, instead of the glass tube inlet for the carrier gas into the detector chamber, stainless steel capillary tubing was used, though the flow was still restricted to 25 ml/min; the second change was that over the end of the detector chamber, a glass plate had been stuck on by araldite so as to obtain a vacuum seal as well as allow the discharge glow to be observed from outside.

It was possible to reduce the pressure in the detector chamber to less than 0.5 mm of mercury without any flow of gas when just connected to a vacuum pump. The electrodes of the "Champion" sparking plug were cleaned with emery paper but otherwise left unaltered. The plug itself was placed on a teflon seating on to the brass cylinder walls of the detector chamber. The actual layout of the system and the electrical circuit was followed as in the original reference[1].

Generally, on increasing the voltage across the electrodes from the stabiliser unit, a blue glow appeared around the electrodes at about 480 V. This was followed by an instantaneous drop in voltage to 360 V and a drop in current. On further decreasing the voltage, the intensity of the discharge was gradually diminished until it finally disappeared at about 230 V. It was over this range that the characteristics of the detector have been reported here.

Fig. 1a shows an increase in the current across the electrodes with a decrease in the degree of vacuum at various constant detector voltages. The degree of vacuum in the detector chamber could be controlled by pinching the rubber tubing between the vacuum pump and the detector. Fig. 1b shows that the current across the detector also increases proportionally to the voltage across the electrodes at various constant pressures inside the detector chamber.

Fig. 2 shows an increase in the sensitivity of the detector when using 5 ml samples of 75 v.p.m. of carbon dioxide in nitrogen at various detector voltages. The sample was injected by means of a sampling valve and it appeared that the detector was most sensitive in the region when the discharge was weakest just before being extinguished at 230 V. The usual set of conditions for Figs. 2 and 3 was as follows:

Carrier gas: high purity nitrogen at 4 p.s.i.g.

Pressure in detector chamber: 2.3 mm of Hg with nitrogen flow

Column: coiled silica gel column for carbon dioxide and carbon monoxide detection

Fig. 3 shows the linearity in response of the detector—measured as peak heights of number of scale divisions on a 1 mV recorder chart of 100 divisions— in a sample

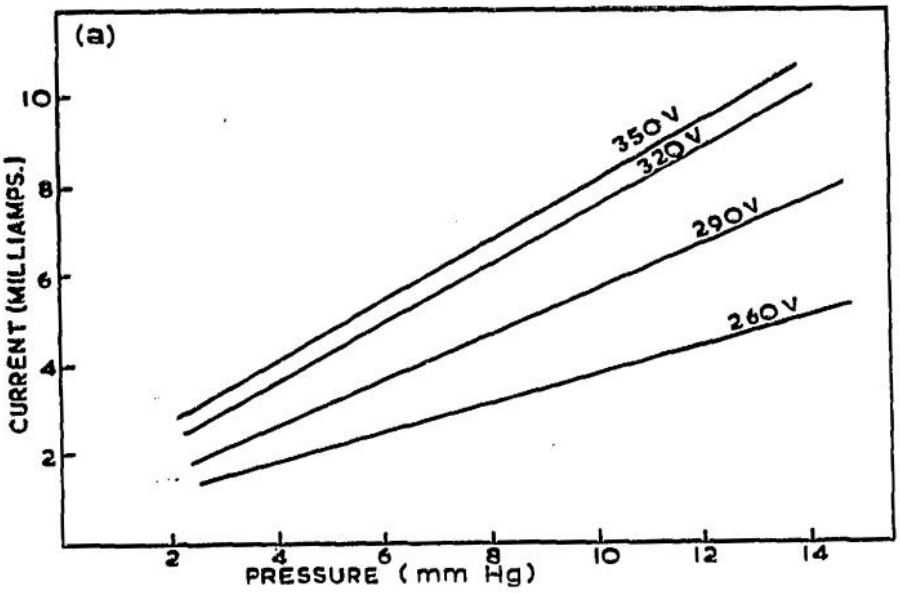

Fig. 1 a. Characteristics of the d.c. detector.

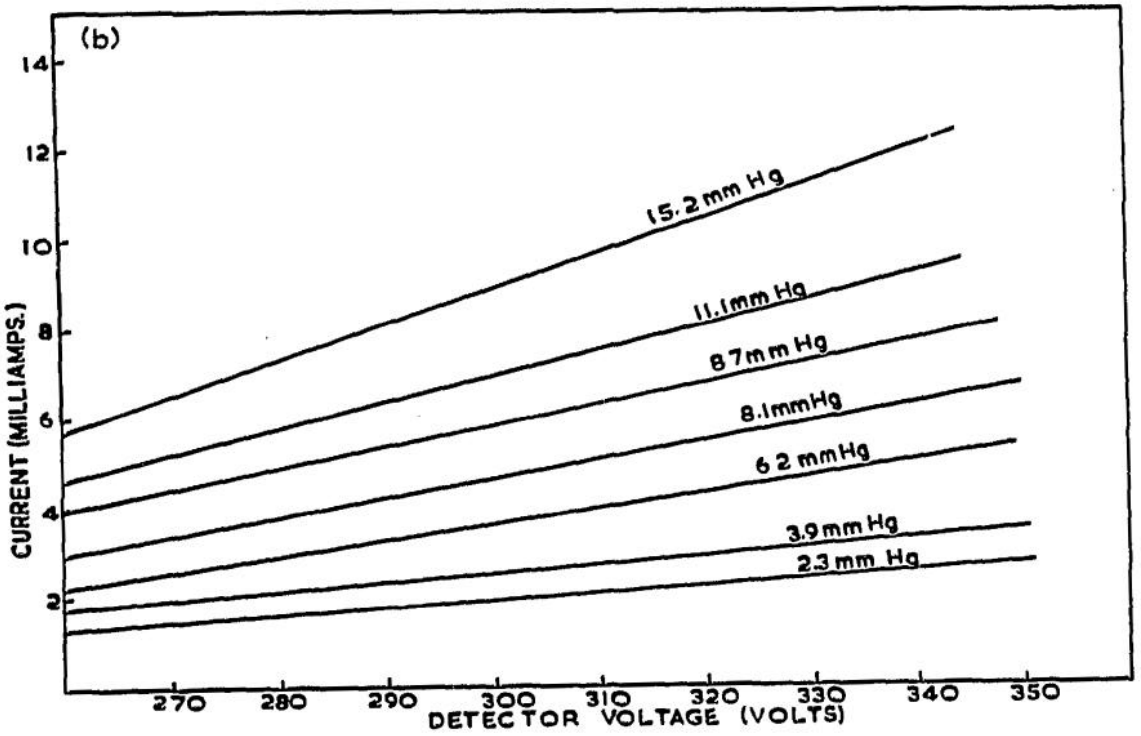

Fig. 1 b. Characteristics of the d.c. discharge detector.

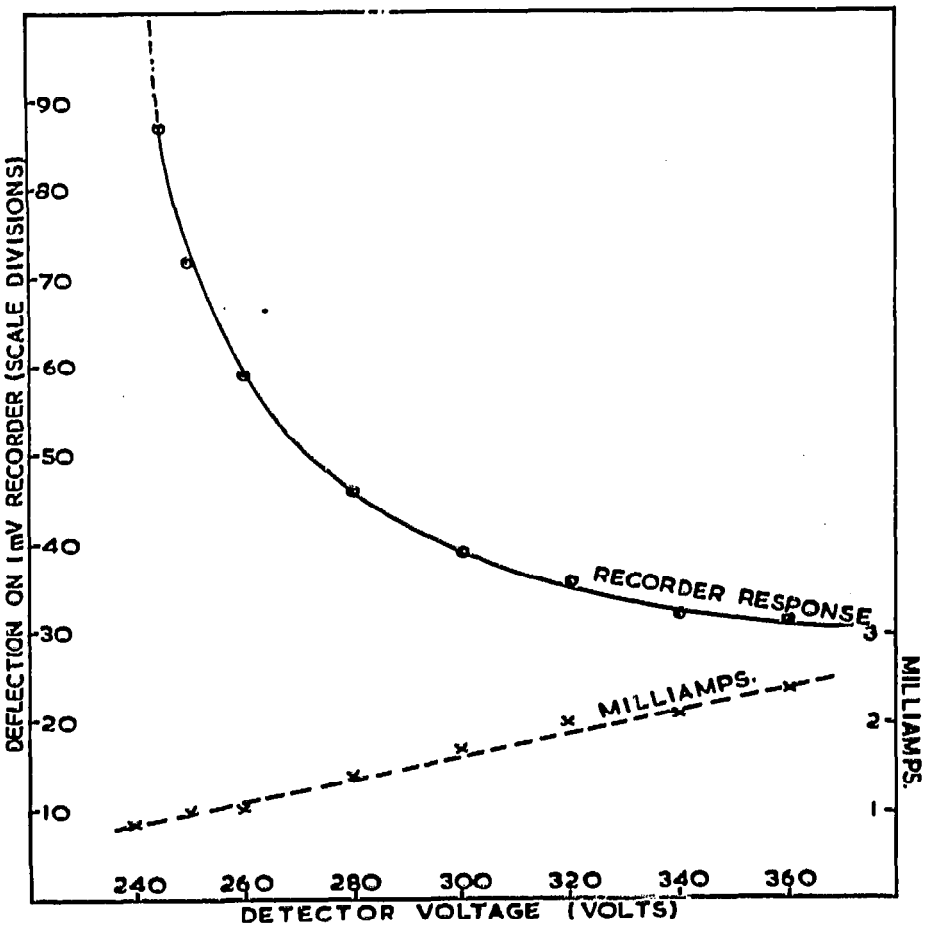

Fig. 2. Recorder response against detector voltage.

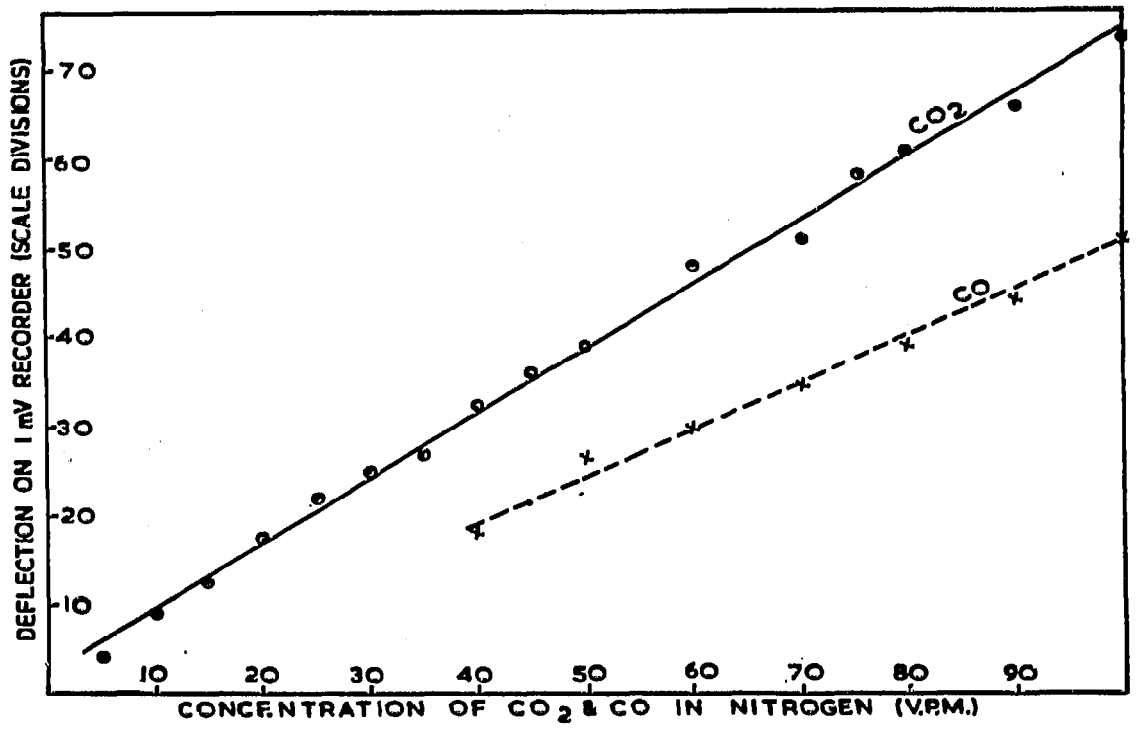

Fig. 3. Linearity in response of d.c. discharge detector.

range from 1 v.p.m. of carbon dioxide in nitrogen to 100 v.p.m. of carbon dioxide in nitrogen and from 40 to 100 v.p.m. of carbon monoxide in nitrogen. For carbon dioxide, the detector voltage was 260 V and a 1 ml sample loop was used; for carbon monoxide, the detector voltage was 235 V and a 5 ml sample loop was used.

The baseline obtained on the 1 mV recorder was very stable, especially at lower voltages when the sensitivity was extremely high. At voltages above 300 V, the detector not only became noisy but hot as well, which caused the seals, used to obtain leak-proof joints, to melt.

The best performance of the detector was obtained at 235 V when a sensitivity of 1 v.p.m. per 4 scale divisions was obtained for carbon dioxide and a sensitivity of 2 v.p.m. per scale division for carbon monoxide (using 5 ml sample sizes). The responses to oxygen and lower hydrocarbon gases were positive but have not been investigated systematically yet. In fact, with a 0.25 ml sample of air, the oxygen content was high enough to extinguish the discharge unless sustained at 350 V or above.

This preliminary work has confirmed that the direct current discharge type detector provides a sensitive means of determining carbon monoxide and carbon dioxide; the response is linear, and under the conditions of greatest sensitivity, the baseline is most stable.

This work was carried out in the Research and Development Centre, The British Oxygen Company, London, S.W. 19.

Research and Development Centre, The British Oxygen Company, London, S.W. 19 (Great Britain)

B. J. RICHARDSON
C. NARAIN

1 E. R. FISHER AND M. MCCARTY, *Anal. Chem.*, 37 (1965) 1208.

Received July 14th, 1966

J. Chromatog., 26 (1967) 248–251

Gas chromatography of diols

The gas chromatography of the even numbered homologues of C_{10}–C_{26} α,ω-diols has been described[1] and some information is available concerning the gas chromatographic separation of lower molecular weight diols[2,3]. This report describes a versatile column packing suitable for the rapid analytical or preparative gas chromatography of homologous and isomeric diols under isothermal conditions.

Experimental

Preparation of the column packing. The solid support was silanized by a modification of the method of BOHEMEN *et al.*[4]. Celite (60–80, 250 g) was added to petroleum ether 60–80 (1.2 l) containing hexamethyldisilazane (62.5 ml) and the mixture refluxed with exclusion of atmospheric moisture for 20 h. The solid was washed with petroleum